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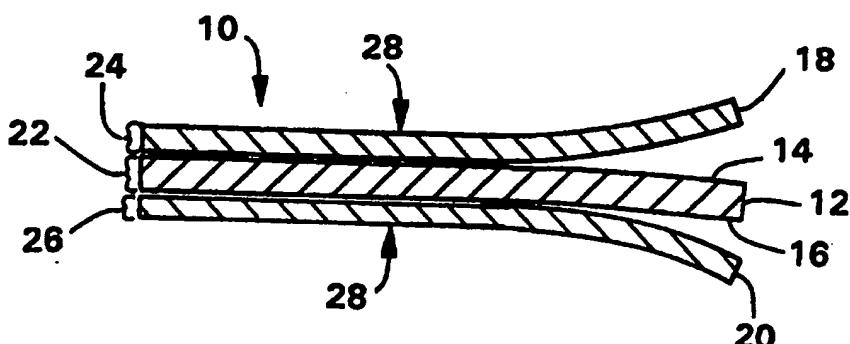
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(71) Applicant: KIMBERLY-CLARK CORPORATION [US/US]; 401 North Lake Street, Neenah, WI 54956 (US).	
(72) Inventors: MCCORMACK, Ann, Louise; 1265 Poplar Grove Lane, Cumming, GA 30131 (US). HETZLER, Kevin, George; 440 Powers Court Avenue, Alpharetta, GA 30201 (US).	Published <i>Without international search report and to be republished upon receipt of that report.</i>
(74) Agents: LEACH, Nicholas, N. et al.; Kimberly-Clark Corporation, 401 North Lake Street, Neenah, WI 54956 (US).	
(54) Title: LOW GAUGE FILMS AND FILM/NONWOVEN LAMINATES	



(57) Abstract

Disclosed herein is a low gauge, multilayer film (10) which may be laminated to other materials such as, for example, fibrous nonwoven webs. The multilayer film may include one or more skin layers (18, 20) which in certain configurations comprise no more than about 15 percent of the overall thickness and in other configurations no more than about 10 percent of the overall thickness of the multilayer film (10). Such films and laminates have a wide variety of uses including, but not limited to, personal care absorbent products, articles of clothing and health care related items such as surgical drapes and gowns.

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## LOW GAUGE FILMS AND FILM/NONWOVEN LAMINATES

5       The present invention is directed to low gauge, multi-layer films with skin layers that are extremely thin.

10      The present invention is directed to extremely thin multi-layer films and their use with laminates. Such materials have a wide variety of uses, especially in the areas of limited use and disposable items.

15      Many products today require highly engineered components and yet, at the same time, these products are required to be limited use or disposable items. By limited use or disposable, it is meant that the product and/or component is used only a small number of times or possibly only once before being discarded. Examples of such products include, but are not limited to, surgical and health care related products such as surgical drapes and gowns, disposable work wear such as coveralls and lab coats and a personal care absorbent products such as diapers, training pants, incontinence garments, sanitary napkins, bandages, wipes and the like. All of these products can and do utilize as components, films and fibrous nonwoven webs. While both materials are often used interchangeably, films tend to have greater barrier properties, especially to liquids while fibrous nonwovens webs have, among other things, better tactile, comfort and aesthetic properties. When these materials are used in limited use and/or disposable products, the impetus for maximizing engineered properties while reducing cost is extremely high. To this end, it is often desirable to use either a film or a nonwoven to achieve the desired results because the combination often becomes more expensive. In the area of films, there have been previous attempts to make multi-layer films with reduced thicknesses. See, for example, U.S. Patent No. 5,261,899 to Vischer wherein a three layer film is made with a central layer that comprises from about 30 to 70% of the total thickness of the multi-layer film. One advantage in forming multi-layer films is that

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specific properties can be designed into the film, and, by making the films multi-layer the more costly ingredients can be relegated to the outer layers where they are most likely to be needed.

5 It is an object of the present invention to provide a multilayer film which can be engineered to provide specific properties while providing such properties in a very thin gauge.

10 The object is solved by the multilayer film according to independent claims 1, 3 and 11, the personal care absorbent article according to independent claims 12 and 16, the laminate according to independent claims 13 and 15, the surgical gown according to independent claims 18 and 19, the surgical drape according to independent claims 20 and 21 and the article of clothing according to independent claims 22 and 23.

15 Further advantages, features, aspects and details of the invention are evident from the dependent claims, the description and the accompanying drawings. The claims are intended to be understood as a first non-limiting approach of defining the invention in general terms.

20 Another aspect of the present invention is to combine such low gauge films with other support layers such as layers of fibrous nonwoven webs to increase strength and provide aesthetic properties.

25 The present invention is directed to multilayer films and multilayer film/nonwoven laminates. In addition, the present invention is directed to such films laminated to other materials such as, for example, fibrous nonwoven webs.

30 The films are made by conventional film forming techniques such as cast and blown coextrusion film forming processes. The films are created with a core layer made from an extrudable thermoplastic polymer with the core layer defining a first exterior surface and a second exterior surface. In the most basic configuration, a first skin layer is attached, usually simultaneously due to the coextrusion process, to the first exterior surface of the core layer to form a multilayer film. The multilayer film defines an overall thickness with the first

skin layer defining a first skin thickness which comprises less than about 10 percent of the overall thickness of the multilayer film. This is due to the stretching of the extruded film to such a degree so as to thin the multilayer film to 5 within the dimensions defined herein. As a result, the thickness of the first skin layer will not exceed about 2  $\mu\text{m}$ . Given the extremely thin nature of the multilayer film, it may be desirable to laminate the multilayer film to another material such as a support layer. Suitable support layers 10 include, but are not limited to, such materials as other films, fibrous nonwoven webs, woven materials, scrims, netting and combinations of the foregoing. In other embodiments of the present invention, the core layer may have a first skin layer attached to the first exterior surface of the core layer and a 15 second skin layer attached to the second exterior surface of the core layer. In such situations, the first skin and the second skin layer should have a combined thickness which does not exceed about 15 percent of the overall thickness and more desirably where neither the first skin thickness nor the second 20 skin thickness exceeds more than about 7.5 percent of the overall thickness of the multilayer film. If desired, one or more of the layers may contain other additives such as, for example, a particulate filler. Most typically, such fillers will be primarily utilized in the core layer in, for example, a 25 weight percent of at least about 60 percent, based upon the total weight of that particular layer.

It is also possible to make multilayer films which are breathable either through the use of specialized polymers which permit diffusion of gases through the layers and/or through the 30 use of particulate fillers. Normally, to make such films breathable, they are stretched and/or crushed between compression rollers so as to create voids in and around the particles to permit the transmission of water vapor and other gases. Typically such breathable multilayer films will have 35 water vapor transmission rates of at least 300 grams per square meter per 24 hours.

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Such films and laminates have a wide variety of uses including, but not limited to, applications in personal care absorbent articles including diapers, training pants, sanitary napkins, incontinence devices, bandages and the like. These same films and laminates also may be used in items such as surgical drapes and gowns as well as various articles of clothing either as the entire article or simply as a component thereof.

The invention will be better understood by reference to the following description of embodiments of the invention taken in conjunction with the accompanying drawings, wherein:

Figure 1 is a cross-sectional side view of a multilayer film according to the present invention. The right side of the film has been split apart to facilitate its description.

Figure 2 is a cross-sectional side view of a multilayer film/nonwoven laminate according to the present invention.

Figure 3 is a schematic side view of a process for forming a multilayer film according to the present invention and a multilayer film/nonwoven laminate according to the present invention.

Figure 4 is a partially cut away top plan view of an exemplary personal care absorbent article, in this case a diaper, which may utilize a multilayer film and multilayer film/nonwoven laminate according to the present invention.

The present invention is directed to multilayer films, that is, films having two or more layers as well as such films laminated to support layers such as, for example, fibrous nonwoven webs. Referring to Figure 1, there is shown, not to scale, a multilayer film 10 which, for purposes of illustration, has been split apart at the right side of the drawing. The multilayer film 10 includes a core layer 12 made from an extrudable thermoplastic polymer such as a polyolefin or a blend of polyolefins. The core layer 12 has a first exterior surface 14 and a second exterior surface 16. The core layer also has a core thickness 22. Attached to the first exterior surface 14 of the core layer 12 is a first skin layer 18 which has a first skin thickness 24. Attached to the second

exterior surface 16 of the core layer 12 is an optional second skin layer 20 which has a second skin thickness 26. In addition, the multilayer film 10 has an overall thickness 28. Such multilayer films 10 can be formed by a wide variety of processes well known to those of ordinary skill in the film forming industry. Two particularly advantageous processes are cast film coextrusion processes and blown film coextrusion processes. In such processes, the two or three layers are formed simultaneously and exit the extruder in a multilayer form. Due to the extremely thin nature of the multilayer films according to the present invention such processes will most likely prove to be the most advantageous though it also may be possible to form multilayer films using separate extrusion processes. For more information regarding such processes, see, for example, U.S. Patent Numbers 4,522,203; 4,494,629 and 4,734,324 which are incorporated herein by reference in their entirety.

An important feature of the present invention is the ability to utilize a more generic core layer 12 in conjunction with a much thinner and more specially designed skin layer such as the first skin layer 18 or a combination of two or more skin layers attached to either or both sides of the core layer 12. Thus, it is possible to form multilayer films 10 with many layers of material. The core layer 12 as with the first skin layer 18 and optional second skin layer 20 may be formed from any polymers which are capable of being utilized in multilayer film constructions including, but not limited to, polyolefins including homopolymers, copolymers, and blends. To further reduce the cost of the core layer 12 one or more types of fillers may be added to the core layer polymer extrusion blend. Both organic and inorganic fillers may be used. The fillers should be selected so as to not chemically interfere with or adversely affect the extruded film. These fillers can be used to reduce the amount of polymer being used for the core layer 12 and/or to impart particular properties such as breathability and/or odor reduction. Examples of fillers can include, but are not limited to, calcium carbonate ( $\text{CaCO}_3$ ), various kinds of

clay, silica ( $\text{SiO}_2$ ), alumina, barium sulfate, sodium carbonate, talc, magnesium sulfate, titanium dioxide, zeolites, aluminum sulfate, cellulose-type powders, diatomaceous earth, magnesium sulfate, magnesium carbonate, barium carbonate, kaolin, mica, 5 carbon, calcium oxide, magnesium oxide, aluminum hydroxide, pulp powder, wood powder, cellulose derivatives, polymer particles, chitin and chitin derivatives.

The amount of filler that can be used resides within the discretion of the end-user, however, additions of from 0 to 80% by weight based upon the total weight of the core layer 12 are possible. Generally the fillers will be in particulate form and usually will have somewhat of a spherical shape with average particle sizes in the range of about 0.1 to about 7  $\mu\text{m}$ . Furthermore, if sufficient filler is used in combination with 15 sufficient stretching of the multilayer film 10, then voids can be created around the particles contained within the core layer 12 thereby making the core layer breathable. High loadings, in excess of about 60 percent by weight of the core layer 12 when combined with stretching provides films which are breathable. 20 Such breathable films will generally have Water Vapor Transmission Rates (WVTR) in excess of 300 grams per square meter per 24 hours.

The skin layers 18 and 20 will typically include extrudable thermoplastic polymers and/or additives which provide specialized properties to the multilayer film 10. Thus, the first skin layer 18 and/or the second skin layer 20 may be made from polymers which give such properties as antimicrobial activity, water vapor transmission, adhesion and/or antiblocking properties. Thus, the particular polymer or polymers chosen for the skin layer 18 and 20 will depend upon 25 the particular attributes desired. Examples of possible polymers that may be used alone or in combination include homopolymers, copolymers and blends of polyolefins as well as ethylene vinyl acetate (EVA), ethylene ethyl acrylate (EEA), 30 ethylene acrylic acid (EAA), ethylene methyl acrylate (EMA), ethylene butyl acrylate (EBA), polyester (PET), nylon (PA), 35

ethylene vinyl alcohol (EVOH), polystyrene (PS), polyurethane PU) and olefinic thermoplastic elastomers which are multistep reactor products wherein an amorphous ethylene propylene random copolymer is molecularly dispersed in a predominately 5 semicrystalline high polypropylene monomer/low ethylene monomer continuous matrix.

Oftentimes it may be desirable to laminate the multilayer film 10 to one or more substrates or support layers 30 such as is shown in Figure 2. The core layer may not have sufficient 10 adhesive or attachment properties so as to make it bondable to the support layer 30. As a result, the first skin layer 18 may be made from a polymer or polymers which exhibit higher adhesive properties and/or a lower tack point than the core layer 12.

A desired result with respect to the material of the present invention is to achieve a very low overall film thickness and more importantly, skin layers which are only a small percentage of the overall thickness of the multilayer film 10. As demonstrated by the examples below, based upon the 15 overall thickness 28 of the multilayer film 10, in two layer constructions the first skin thickness 24 of the first skin layer 18 should not exceed more than 10 percent of the overall thickness 28. In three layer film constructions the combined thickness of the first skin layer 18 and second skin layer 20 should not exceed 15 percent of the overall thickness and generally, the first skin layer 18 should not exceed more than 20 25 7.5 percent of the overall skin thickness 28. The same is also true with respect to the second skin layer 20. As a result, the core thickness 22 comprises at least 85 percent of the overall thickness 28 and the first skin layer 18 and second skin layer 20 each generally will comprise no more than 7.5 percent of the overall thickness 28. Generally, it has been 30 possible to create thinned films with overall thicknesses 28 of 30  $\mu\text{m}$  or less and in certain applications with skin layers that do not exceed two  $\mu\text{m}$ . This is made possible by first forming a 35 multilayer film 10 and then stretching or orienting the film in

the machine direction, as explained in greater detail below, such that the resultant multilayer film 10 has increased strength properties in the machine direction, i.e., the direction which is parallel to the direction of the film as it 5 is taken off the film extrusion equipment.

The resultant film can, if desired, be laminated to one or more support layers 30 as are shown in Figure 2. The support layers 30 as shown in Figure 2 are fibrous nonwoven webs. The manufacture of such fibrous nonwoven webs is well known to 10 those of ordinary skill in the art of nonwoven manufacturing. Such fibrous nonwoven webs can add additional properties to the multilayer film 10 such as, a more soft, cloth-like feel. This is particularly advantageous when the multilayer film 10 is being used as a barrier layer to liquids in such applications 15 as outer covers for personal care absorbent articles and as barrier materials for hospital, surgical, and clean room applications such as, for example, surgical drapes, gowns and other forms of apparel. Attachment of the support layers 30 to the first skin layer 18 and second skin layer 20 may be by the 20 use of a separate adhesive such as hot-melt and solvent based adhesives or through the use of heat and/or pressure as with heated bonding rolls. As a result, it may be desirable to design either or both the first skin layer 18 and the second skin layer 20 so as to have inherent adhesive properties to 25 facilitate the lamination process.

A particularly advantageous support layer is a fibrous nonwoven web. Such webs may be formed from a number of processes including, but not limited to, spunbonding, meltblowing and bonded carded web processes. Meltblown fibers 30 are formed by extruding molten thermoplastic material through a plurality of fine, usually circular, die capillaries as molten threads or filaments into a high velocity usually heated gas stream such as air, which attenuates the filaments of molten thermoplastic material to reduce their diameters. Thereafter, 35 the meltblown fibers are carried by the high velocity usually heated gas stream and are deposited on a collecting surface to form a web of randomly dispersed meltblown fibers. The

meltblown process is well-known and is described in various patents and publications, including NRL Report 4364, "Manufacture of Super-Fine Organic Fibers" by B. A. Wendt, E. L. Boone and C. D. Fluharty; NRL Report 5265, "An Improved 5 Device For The Formation of Super-Fine Thermoplastic Fibers" by K. D. Lawrence, R. T. Lukas, J. A. Young; U.S. Patent Number 3,676,242, issued July 11, 1972, to Prentice; and U.S. Patent Number 3,849,241, issued November 19, 1974, to Buntin, et al. The foregoing references are incorporated herein by reference 10 in their entirety.

Spunbond fibers are formed by extruding a molten thermoplastic material as filaments from a plurality of fine, usually circular, capillaries in a spinnerette with the diameter of the extruded filaments then being rapidly reduced, 15 for example, by non-eductive or eductive fluid-drawing or other well-known spunbonding mechanisms. The production of spunbond nonwoven webs is illustrated in patents such as Appel et al., U.S. Patent Number 4,340,563; Matsuki, et al., U.S. Patent Number 3,802,817; Dorschner et al., U.S. Patent 3,692,618; 20 Kinney, U.S. Patent Numbers 3,338,992 and 3,341,394; Levy, U.S. Patent Number 3,276,944; Peterson, U.S. Patent Number 3,502,538; Hartman, U.S. Patent Number 3,502,763; Dobo et al., U.S. Patent Number 3,542,615; and Harmon, Canadian Patent Number 803,714. All of the foregoing references are 25 incorporated herein by reference in their entirety.

Multilayer support layers 30 also may be used. Examples of such materials can include, for example, spunbond/meltblown laminates and spunbond/meltblown/spunbond laminates such as are taught in Brock et al., U.S. Patent Number 4,041,203 which is 30 incorporated herein by reference in its entirety.

Bonded carded webs are made from staple fibers which are usually purchased in bales. The bales are placed in a picker which separates the fibers. Next the fibers are sent through a combing or carding unit which further breaks apart and aligns 35 the staple fibers in the machine direction so as to form a machine direction-oriented fibrous nonwoven web. Once the web has been formed, it is then bonded by one or more of several

bonding methods. One bonding method is powder bonding wherein a powdered adhesive is distributed throughout the web and then activated, usually by heating the web and adhesive with hot air. Another bonding method is pattern bonding wherein heated calender rolls or ultrasonic bonding equipment is used to bond the fibers together, usually in a localized bond pattern though the web can be bonded across its entire surface if so desired.

When using bicomponent staple fibers, through-air bonding equipment is, for many applications, especially advantageous.

A process for forming the multilayer film 10 is shown in Figure 3 of the drawings. Referring to the figure, the multilayer film 10 is formed from a coextrusion film apparatus 40 such as a cast or blown unit as was previously described above. Typically the apparatus 40 will include two or more polymer extruders 41. The multilayer film 10 is extruded into a pair of nip or chill rollers 42 one of which may be patterned so as to impart an embossed pattern to the newly formed film 10. This is particularly advantageous to reduce the gloss of the film and give it a matte finish. Using a three layer film construction such as is shown in Figure 1, typically the multilayer film 10, as initially formed, will have an overall thickness 28 of approximately 40  $\mu\text{m}$  or greater with the first skin layer 18 and the second skin layer 20 each having initial thicknesses of 3  $\mu\text{m}$  or greater which collectively is approximately 15% of the overall initial thickness.

From the coextrusion film apparatus 40 the film 10 is directed to a film stretching unit 44 such as a machine direction orienter which is a commercially available device from vendors such as the Marshall and Williams Company of Providence, Rhode Island. Such an apparatus 44 has a plurality of stretching rollers 46 which progressively stretch and thin the multilayer film 10 in the machine direction of the film which is the direction of travel of the film 10 through the process as shown in Figure 3. After exiting the film stretching unit 44 the film 10 should have a maximum thickness of approximately 30  $\mu\text{m}$  and each of the skin layers should have

a maximum thickness of no more than about 2  $\mu\text{m}$  which in turn is collectively less than about 15 percent of the overall film and more desirably less than 10 percent of the overall film thickness.

5        If desired, the multilayer film 10 may be attached to one or more support layers 30 to form a multilayer film/nonwoven laminate 32. Referring again to Figure 3, a conventional fibrous nonwoven web forming apparatus 48, such as a pair of spunbond machines, is used to form the support layer 30. The  
10 long, essentially continuous fibers 50 are deposited onto a forming wire 52 as an unbonded web 54 and the unbonded web 54 is then sent through a pair of bonding rolls 56 to bond the fibers together and increase the tear strength of the resultant web support layer 30. One or both of the rolls are often heated to aid in bonding. Typically, one of the rolls 56 is  
15 also patterned so as to impart a discrete bond pattern with a prescribed bond surface area to the web 30. The other roll is usually a smooth anvil roll but this roll also may be patterned if so desired. Once the multilayer film 10 has been  
20 sufficiently thinned and oriented and the support layer 30 has been formed, the two layers are brought together and laminated to one another using a pair laminating rolls or other means 58.  
As with the bonding rolls 56, the laminating rolls 58 may be heated. Also, at least one of the rolls may be patterned to  
25 create a discrete bond pattern with a prescribed bond surface area for the resultant laminate 32. Generally, the maximum bond point surface area for a given area of surface on one side of the laminate 32 will not exceed about 50 percent of the total surface area. There are a number of discrete bond patterns which may be used. See, for example, Brock et al.,  
30 U.S. Patent Number 4,041,203 which is incorporated herein by reference in its entirety. Once the laminate 32 exits the laminating rolls 58, it may be wound up into a roll 60 for subsequent processing. Alternatively, the laminate 32 may  
35 continue in-line for further processing or conversion.

The process shown in Figure 3 also may be used to create a three layer laminate 32 such as is shown in Figure 2 of the drawings. The only modification to the previously described process is to feed a supply 62 of a second fibrous nonwoven web support layer 30 into the laminating rolls 58 on a side of the multilayer film 10 opposite that of the other fibrous nonwoven web support layer 30. As shown in Figure 3, the supply of support layer 30 is in the form of a preformed roll 62. Alternatively, as with the other layers, the support layer 30 may be formed directly in-line. In either event, the second support layer 30 is fed into the laminating rolls 58 and is laminated to the multilayer film 10 in the same fashion as the other support layer 30.

As has been stated previously, the multilayer film 10 and the laminate 32 may be used in a wide variety of applications not the least of which includes personal care absorbent articles such as diapers, training pants, incontinence devices and feminine hygiene products such as sanitary napkins. An exemplary article 80, in this case a diaper, is shown in Figure 4 of the drawings. Referring to Figure 4, most such personal care absorbent articles 80 include a liquid permeable top sheet or liner 82, a back sheet or outercover 84 and an absorbent core 86 disposed between and contained by the top sheet 82 and back sheet 84. Articles 80 such as diapers may also include some type of fastening means 88 such as adhesive fastening tapes or mechanical hook and loop type fasteners.

The multilayer film 10 by itself or in other forms such as the multilayer film/support layer laminate 32 may be used to form various portions of the article including, but not limited to, the top sheet 82 and the back sheet 84. If the film is to be used as the liner 82, it will most likely have to be apertured or otherwise made to be liquid permeable. When using a multilayer film/nonwoven laminate 32 as the outercover 84, it is usually advantageous to place the nonwoven side facing out away from the user. In addition, in such embodiments it may be possible to utilize the nonwoven portion of the laminate 32 as the loop portion of the hook and loop combination.

Other uses for the multilayer film and multilayer film/support layer laminates according to the present invention include, but are not limited to, surgical drapes and gowns, wipers, barrier materials and articles of clothing or portions thereof including such items as workwear and lab coats.

The properties of the present invention were determined using a series of test procedures which are set forth below. These properties include film thicknesses, water vapor transmission rates and peel strengths.

10

#### Water Vapor Transmission Rate

The water vapor transmission rate (WVTR) for the sample materials was calculated in accordance with ASTM Standard E96-15 80. Circular samples measuring 7.62 cm (three inches) in diameter were cut from each of the test materials and a control which was a piece of CELGARD® 2500 film from Hoechst Celanese Corporation of Sommerville, New Jersey. CELGARD® 2500 film is a microporous polypropylene film. Three samples were prepared 20 for each material. The test dish was a number 60-1 Vapometer pan distributed by Thwing-Albert Instrument Company of Philadelphia, Pennsylvania. One hundred milliliters of water was poured into each Vapometer pan and individual samples of the test materials and control material were placed across the 25 open tops of the individual pans. Screw-on flanges were tightened to form a seal along the edges of each pan, leaving the associated test material or control material exposed to the ambient atmosphere over a 6.5 centimeter diameter circle having an exposed area of approximately 33.17 square centimeters. The 30 pans were placed in a forced air oven at 32°C (100°F). The oven was a constant temperature oven with external air circulating through it to prevent water vapor accumulation inside. A suitable forced air oven is, for example, a Blue M Power-O-Matic 60 oven distributed by Blue M Electric Company of Blue 35 Island, Illinois. After 24 hours, the pans were removed from

the oven and weighed again. The preliminary test water vapor transmission rate values were calculated as follows:

5           Test WVTR = (grams weight loss over 24 hours) x 315.5  
              g/m<sup>2</sup> / 24 hrs

The relative humidity within the oven was not specifically controlled.

10          Under predetermined set conditions of 32°C (100°F) and ambient relative humidity, the WVTR for the CELGARD® 2500 control has been determined to be 5000 grams per square meter for 24 hours. Accordingly, the control sample was run with each test and the preliminary test values were corrected to set 15 conditions using the following equation:

20           WVTR = (Test WVTR/control WVTR) x 5000 g/m<sup>2</sup>/24 hrs.)  
              (g/m<sup>2</sup> / 24 hrs)

#### Film/Film Layer Thicknesses

25          The overall thickness 28, first skin thickness 24, core thickness 22 and second skin thickness 26 were measured in cross-section by Field Emission Scanning Electron Microscopy (FESEM). Each film sample was submersed in liquid nitrogen and cut on impact with a razor blade. The freshly cut cross-30 section was mounted to a specimen stub in an upright position using copper tape. The samples were observed using an Hitachi S-800 Field Emission Scanning Electron Microscope at 5 and 10 keV. Scanning electron photomicrographs were taken at 2000X magnification to show the film structures for each sample.  
35          Three separate samples and corresponding pictures were prepared for each multilayer film. The 10.2 centimeter by 12.7 centimeter negatives were enlarged to 20.4 centimeter by 25.4 centimeter copies and the measurements were taken directly off these photographs. A reference 15 µm scale magnified at 2000X. 40          was superimposed on each photo. Five measurements for each of

the layers was made on each of the three photos for each film sample thereby creating 15 datapoints or measurements for each thickness. Measurements were made for the first skin layer, the second skin layer and the core layer of each sample. The 5 15 measurements for each layer were combined and averaged to yield a thickness value in  $\mu\text{m}$  for each layer. The total thickness of the overall film was obtained by adding the average values for the core layer, first skin layer and second skin layer for each sample. The relative percentage of each 10 skin layer was obtained by dividing the average thickness of the respective skin layer by the average overall thickness of the same sample and multiplying the result by 100 to yield percent.

15 180° T Peel Test

To test the bond strength between the film layer and the fibrous nonwoven comfort layer, a delamination or peel strength test was performed upon samples of the various materials. 10.2 20 centimeter (cm) by approximately 15.2 centimeter (cm) samples of the material were cut. To the film side of the samples there was applied a 10.2 cm by 15.2 cm piece of 3M 2308 masking tape. The sample were then rolled, tape side up, forward and backwards two times with a 22.2 kilogram roller weight. The 25 samples were then manually delaminated at one of the short ends to produce edges which could be placed within the jaws of a Sintech®/2 Computer Integrated Testing System manufactured by MTS Systems Corporation of Eden Prairie, MN. The jaw gap was set at a span of 100 millimeters and enough of the material was 30 left in the laminated state so that the jaws could travel 65 millimeters. The sample was positioned in the jaws so that the sample would start delaminating before the jaws had been expanded 10 millimeters. The crosshead speed was set at 300 millimeters per minute and the data were then recorded between 35 the 10 millimeter start point and the 65 millimeter end point.

The data recorded indicated the peel strength or load

necessary in grams to separate the two layers and the standard index in grams with a maximum, minimum and mean value.

All the example films were three layer films with the two outer or skin layers in each example being the same. All films  
5 were cast films and all the films were embossed prior to stretching to yield a matte finish on the films. In addition, all the films were laminated to a 17 gram per square meter (gsm) polypropylene spunbond web made from approximately 0.22 tex (2 denier) fibers. The spunbond web was prebonded with a  
10 point bond pattern having an overall bond area of approximately 15 percent.

#### Example 1

15 In Example 1 the core layer was, on a weight percent basis based upon the total weight of the layer, 65 percent ECC English China Supercoat™ calcium carbonate with a 1  $\mu\text{m}$  average particle size and a 7  $\mu\text{m}$  top cut. The calcium carbonate was obtained from ECCA Calcium Products, Inc. of Sylacauga, Alabama which is a division of ECC International. The core layer also included 15 percent Exxon 9302 Random Copolymer Polypropylene (RCP) from the Exxon Chemical Company of Houston, Texas, 15 percent Himont KS059 Catalloy polymer from Himont U.S.A. of Wilmington, Delaware and 5 percent Quantum NA206 Low Density  
20 Polyethylene (LDPE) from Quantum Chemical Corporation of New York, New York. The Himont Catalloy polymer is an olefinic thermoplastic elastomer or TPO multistep reactor product wherein an amorphous ethylene propylene random copolymer is molecularly dispersed in a predominately semicrystalline high  
25 polypropylene monomer/low ethylene monomer continuous matrix.  
30

The two outer or skin layers on opposite sides of the core layer comprised 15 percent Himont KS057 Catalloy polymer from Himont U.S.A., 20 percent Ampacet 10115 antiblock and 65 percent Exxon XC-101 (28 percent EMA copolymer). Ampacet 10115  
35 antiblock comprises 20 weight percent Superfloss™ diatomaceous earth let down in 79.75 percent Chevron 2207 EMA and 0.25

percent aluminum stearate. Ampacet 10115 antiblock is available from Ampacet Corporation of Tarrytown, New York. Chevron 2207 EMA is available from the Chevron Chemical Corporation of San Ramon, California and Exxon XC-101 is 5 available from the Exxon Chemical Company of Houston, Texas.

The three layer film was extruded using cast extrusion equipment of the type described above. The exiting melt temperature for the skin layers was measured to be 196°C and for the core was 223°C. The air gap (distance between the die 10 and the nip chill roll) was 53 centimeters and the gauge of the resultant film was 38  $\mu\text{m}$  (microns). The film was wound up on a roll and later sent through a Machine Direction Orienter (MDO) Model No. 7200 from the Marshall and Williams Company of Providence, Rhode Island. The MDO unit was preheated to 77°C 15 and the film was stretched 4X while at a temperature of 77°C. By saying the film was stretched 4X it is meant that, for example, a 1 meter long film would be stretched to a resultant length of 4 meters. The final measured thickness of the film 20 was 16.08  $\mu\text{m}$  and the basis weight was 17 grams per square meter (gsm). Each of the two skin layers comprised approximately 2.7 percent of the overall film thickness. As a result, the core layer represented 94.6 percent of the overall thickness. Using the film thickness measuring method described above, the two skin layers each had a film thickness of 0.44  $\mu\text{m}$  and the core 25 layer had a thickness of 15.2  $\mu\text{m}$ .

The resultant film was then thermally laminated to the above-described spunbond layer using a patterned bonding roll having a temperature of approximately 77°C and a smooth anvil roll at a temperature of approximately 54°C with a nip pressure 30 of 4,218 kilograms per meter ( $\text{kg}/\text{m}$ ) while at a line speed of 152 meters per minute with an overall bond area of 15 percent based upon the surface area of one side of the film. The laminate was passed through the bonder in such a fashion that the spunbond layer was adjacent the pattern roll and the film 35 layer was adjacent the smooth anvil roll. The resultant

laminate had a Water Vapor Transmission Rate (WVTR) measured as described above of 2570 grams per square meter per 24 hours ( $2570\text{g}/\text{m}^2/\text{24hr}$  or day). The laminate had a hydrohead of 70 centimeters and a peel strength of 48 grams.

5

Example 2

In Example 2 the core layer was, on a weight percent basis based upon the total weight of the layer, 65 percent ECC 10 English China Supercoat™ calcium carbonate, 15 percent Exxon 9302 Random Copolymer Polypropylene (RCP), 15 percent Himont KS059 Catalloy polymer and 5 percent Quantum NA206 Low Density Polyethylene (LDPE).

The two outer or skin layers on opposite sides of the core 15 layer comprised 15 percent Ampacet 10115 antiblock (20 percent diatomaceous earth antiblock let down in a 24 percent copolymer ethylene methyl acrylate (EMA)) concentrate or masterbatch and 85 percent Himont KS057 Catalloy polymer.

The three layer film was extruded using cast extrusion 20 equipment of the type described above. The exiting melt temperature from the extruder for the skin layers was  $188^\circ\text{C}$  and the melt temperature for the core layer was  $223^\circ\text{C}$ . The air gap (distance between the die heads and the forming nip) was 53 centimeters and the gauge of the resultant film was  $35.5\ \mu\text{m}$ .

The film was wound up on a roll and later sent through the MDO 25 unit which was preheated to  $88^\circ\text{C}$  and the film was stretched 3X while at a temperature of  $88^\circ\text{C}$ . The final measured thickness of the film was  $17.28\ \mu\text{m}$  and the basis weight was 15 gsm. Each 30 of the two skin layers comprised 2 percent of the overall film thickness. As a result, the core layer represented 96 percent of the overall thickness. Using the film thickness measuring method described above, the two skin layers each had a film thickness of  $0.33\ \mu\text{m}$ (microns) and the core layer had a thickness of  $16.62\ \mu\text{m}$ .

The resultant film was then thermally laminated to the above-described spunbond layer using a patterned bonding roll having a temperature of approximately 77°C and a smooth anvil roll at a temperature of approximately 54°C with a nip pressure of 4,218 kg/m while at a line speed of 152 meters per minute with an overall bond area of 15 to 18 percent based upon the surface area of one side of the film. The laminate was passed through the bonder in such a fashion that the spunbond layer was adjacent the pattern roll and the film layer was adjacent the anvil roll. The film had a Water Vapor Transmission Rate (WVTR) measured as described above of 925 grams per square meter per 24 hours and the laminate had a WVTR of 820g/m<sup>2</sup>/24hr. The laminate had a hydrohead of 113 centimeters and a peel strength of 62 grams.

15

Example 3

In example 3 the core layer was, on a weight percent basis based upon the total weight of the layer, 63 percent ECC English China Supercoat™ calcium carbonate, 19 percent Himont KS059 Catalloy polymer, 13 percent Shell 6D81 polypropylene from the Shell Chemical Company of Houston, Texas and 5 percent Dow 4012 low density polyethylene (LDPE) from Dow Chemical U.S.A. of Midland, Michigan.

25

The two outer or skin layers on opposite sides of the core layer comprised 30 percent Himont KS057 Catalloy polymer, 20 percent Techmer S110128E62 antiblock/EVA concentrate or masterbatch from Techmer PM of Rancho Dominguez, California, 20 percent Exxon 760.36 EMA and 30 percent Lotryl 29MA03 Ester-Modified EMA copolymer from Elf Atochem N.A. of Philadelphia, Pennsylvania.

30

The three layer film was extruded using cast extrusion equipment. The exiting temperature from the extruder for the skin layers was 188°C and for the core was 209°C. The air gap (distance between the die heads and the forming roll) was 66 centimeters and the gauge of the resultant film was 38 µm. The

film was wound up on a roll and later sent through the MDO unit which was preheated to 71°C and the film was stretched 4X while at a temperature of 71°C and subsequently annealed at a temperature of approximately 85°C.

5       The final gauge of the film was 16.98 µm and the basis weight was 17 gsm. Each of the two skin layers comprised 3.7 percent of the overall film thickness. As a result, the core layer represented 92.6 percent of the overall thickness. Using the film thickness measuring method described above, the two  
10      skin layers each had a film thickness of 0.6 µm and the core layer had a thickness of 15.77 µm.

The resultant film was then thermally laminated to the above-described spunbond layer using a patterned bonding roll having a temperature of approximately 110°C and a smooth anvil  
15      roll at a temperature of approximately 32°C with a nip pressure of 4570 kg/m 2.54 cm (inch) while at a line speed of 61 meters per minute with an overall bond area of 15 to 18 percent based upon the per unit surface area of one side of the film. The laminate was passed through the bonder in such a fashion that  
20      the spunbond layer was adjacent the pattern roll and the film layer was adjacent the anvil roll. The film had a Water Vapor Transmission Rate (WVTR) measured as described above of 1301 grams per square meter per 24 hours (1301g/m<sup>2</sup>/24hr) and the resultant laminate had a WVTR of 1184g/m<sup>2</sup>/24hr. The laminate  
25      had a hydrohead of 110 centimeters and a peel strength of 161 grams.

Example 4

In Example 4 the core layer was, on a weight percent basis  
30      based upon the total weight of the layer, 63 percent ECC English China Supercoat™ calcium carbonate, 19 percent Himont KS059 Catalloy polymer, 13 percent Shell 6D81 polypropylene and 5 percent Dow 4012 LDPE.

The two outer or skin layers on opposite sides of the core  
35      layer comprised 35 percent Himont KS057 Catalloy polymer, 20

percent Techmer S110128E62 antiblock/EVA  
concentrate/masterbatch and 45 percent Exxon 760.36 EMA.

The three layer film was extruded using cast extrusion equipment. The exiting temperature for the skin layers was  
5 187°C and for the core was 208°C. The air gap (distance between the die heads and the forming roll) was 66 centimeters and the gauge of the resultant film was 35.5 µm. The film was wound up on a roll and later sent through the MDO unit which was preheated to 71°C and the film was stretched 4X while at a  
10 temperature of 71°C and subsequently annealed at a temperature of approximately 85°C. The final measured thickness of the film was 15.96 µm and the basis weight was 15 gsm. Each of the two skin layers comprised 3.0 percent of the overall film thickness. As a result, the core layer represented 94.0  
15 percent of the overall thickness. Using the film thickness measuring method described above, the two skin layers each had a film thickness of 0.48 µm and the core layer had a thickness of 15.0 µm.

The resultant film was then thermally laminated to the  
20 above-described spunbond layer using a patterned bonding roll having a temperature of approximately 110°C and a smooth anvil roll at a temperature of approximately 66°C with a nip pressure of 4570 kg/m while at a line speed of 61 meters per minute with and overall bond area of 15 to 18 percent based upon the per  
25 unit surface area of one side of the film. The laminate was passed through the bonder in such a fashion that the spunbond layer was adjacent the pattern roll and the film layer was adjacent the anvil roll. The resultant laminate had a WVTR of 1522 g/m<sup>2</sup>/24 hrs., a hydrohead of 89 centimeters and a peel  
30 strength of 148 grams.

#### Example 5

In Example 5 the core layer was, on a weight percent basis  
35 based upon the total weight of the layer, 65 percent ECC

English China Supercoat™ calcium carbonate, 15 percent Himont KS059 Catalloy polymer, 15 percent Exxon 9302 Random Copolymer Polypropylene (RCP) and 5 percent Dow 4012 LDPE.

The two outer or skin layers on opposite sides of the core 5 layer comprised 35 percent Himont KS057 Catalloy polymer, 20 percent Techmer S110128E62 antiblock/EVA concentrate/masterbatch and 45 percent Exxon 760.36 EMA.

The three layer film was extruded using cast extrusion equipment. The exiting temperature for the skin layers was 10 175°C and for the core was 234°C. The air gap (distance between the die heads and the forming roll) was 33 centimeters and the gauge of the resultant film was 35.5 µm. The film was wound up on a roll and later sent through the MDO unit which was preheated to 77°C and the film was stretched 4X while at a 15 temperature of 77°C and subsequently annealed at a temperature of approximately 75°C. The final measured thickness of the film was 16.92 µm and the basis weight was 15 gsm. Each of the two skin layers comprised 1.0 percent of the overall film thickness. As a result, the core layer represented 98.0 20 percent of the overall thickness. Using the film thickness measuring method described above, the two skin layers each had a film thickness of 1.75 µm and the core layer had a thickness of 16.57 µm.

The resultant film was then thermally laminated to the 25 above-described spunbond layer using a patterned bonding roll having a temperature of approximately 73°C and a smooth anvil roll at a temperature of approximately 51°C with a nip pressure of 4218 kg/m while at a line speed of 152 meters per minute with an overall bond area of 15 to 18 percent based upon the 30 per unit surface area of one side of the film. The laminate was passed through the bonder in such a fashion that the spunbond layer was adjacent the pattern roll and the film layer was adjacent the anvil roll. The resultant laminate had a WVTR

of 1930 g/m<sup>2</sup>/24 hrs., a hydrohead of 66 centimeters and a peel strength of 116 grams.

As shown by the above examples of the present invention, very low thickness (less than 30 µm) multilayer films can be 5 formed. In addition, such films can have very thin skin layers which can impart a wide variety of functionalities including making the film vapor permeable, liquid impermeable and adhesive in nature. Furthermore such films can be attached to other support layers such as nonwoven to form laminates.

Having thus described the invention in detail, it should be apparent that various modifications can be made in the present invention without departing from the spirit and scope of the following claims.

CLAIMS:

1. A multilayer film comprising:

a core layer (12) made from an extrudable thermoplastic polymer, said core layer (12) having a first exterior surface (14) and a second exterior surface (16),

5 a first skin layer (18) attached to said first exterior surface (14) of said core layer (12) to form said multilayer film (10),

10 said multilayer film (10) defining an overall thickness (28), and said first skin layer (18) defining a first skin thickness (24), said first skin thickness (24) comprising less than about ten percent of said overall thickness (28), said overall thickness (28) not exceeding about 30  $\mu\text{m}$ .

2. The multilayer film of claim 1 wherein said first skin thickness (24) of said first skin layer (18) does not exceed about 2  $\mu\text{m}$ .

3. A multilayer film especially according to claim 1 or 2 comprising:

5 a core layer (12) made from an extrudable thermoplastic polymer, said core layer (12) having a first exterior surface (14) and a second exterior surface (16),

a first skin layer (18) attached to said first exterior surface (14) of said core layer (12) and a second layer (20) attached to said second exterior surface (16) of said core layer (12) to form said multilayer film (10),

10 said multilayer film (10) defining an overall thickness (28), said first skin layer (18) defining a first skin thickness (24), said second skin layer (20) defining a second skin thickness (26) and said core layer (12) defining a core thickness (22), said first skin thickness (24) comprising not more than about 7.5 percent of said overall thickness (28), said overall thickness (28) not exceeding about 30  $\mu\text{m}$ .

4. The multilayer film of claim 3 wherein said second skin thickness (26) of said second skin layer (20) comprises not more than about 7.5 percent of said overall thickness (28).

5. The multilayer film of claim 3 or 4 wherein said first skin thickness (26) of said first skin layer (18) does not exceed about two  $\mu\text{m}$ .

6. The multilayer film of one of claims 3 to 5 wherein said second skin thickness (26) of said second skin layer (20) does not exceed about two  $\mu\text{m}$ .

7. The multilayer film of one of claims 3 to 6 wherein said first skin thickness (24) of said first skin layer (18) and said second skin thickness (26) of said second skin layer (20) when combines do not exceed more than about fifteen percent of said overall thickness (28).

8. The multilayer film of one of the preceding claims wherein said core layer (12) further contains a particulate filler, said particulate filler being present in said core layer (12) in a weight percent of at least 60 percent based upon the total weight of said core layer (12).

9. The multilayer film of one of the preceding claims wherein said core layer (12) has a plurality of voids.

10. The multilayer film of one of the preceding claims wherein said multilayer film (10) has a water vapor transmission rate of at least 300 grams per square meter per 24 hours.

11. A multilayer film especially according to one of the preceding claims comprising:

a core layer (12) made from an extrudable thermoplastic polymer, said core layer (12) having a first exterior surface (14) and a second exterior surface (16),  
5  
a first skin layer (18) attached to said first exterior surface (14) of said core layer (12) and a second skin layer (20) attached to said second exterior surface (16) of said core layer (12) to form said multilayer film (10),  
10  
said multilayer film (10) defining an overall thickness (28), said first skin layer (18) defining a first skin thickness (24) of about two  $\mu\text{m}$  or less, said second skin layer (20) defining a second skin thickness (26) of about two  $\mu\text{m}$  or less and said core layer (12) defining a core thickness (22),  
15  
said first skin thickness (24) comprising not more than about 7.5 percent of said overall thickness (28) and said second skin thickness (26) comprising not more than about 7.5 percent of said overall thickness (28), said overall thickness (28) not exceeding about 30  $\mu\text{m}$ , said core layer (12) containing a  
20  
filler, said filler being present in said core layer (12) in a weight percent of at least 60 percent based upon the total weight of said core layer (12) and wherein said multilayer film (10) has a water vapor transmission rate of at least 300 grams per square meter per 24 hours.

12. A personal care absorbent article (80) comprising a liquid permeable top sheet (82) and a back sheet (84) with an absorbent core (86) disposed therebetween, at least one of said back sheet (84) and said top sheet (82) including the  
5 multilayer film of one of claims 1 to 11.

13. A laminate comprising:

the multilayer film (10) of one of claims 1 to 11 attached to a support layer (30).

14. The laminate of claim 13 wherein said support layer (30) is a fibrous nonwoven web.

15. A laminate especially according to claim 13 or 14 comprising the multilayer film of one of claims 3 to 11 wherein separate support layers are attached to each of said first skin layer and said second skin layer.

16. A personal care absorbent article (80) comprising a liquid permeable top sheet (82) and a back sheet (84) with an absorbent core (86) disposed therebetween, at least one of said back sheet (84) and said top sheet (82) including the laminate of one of claims 13 to 15.

17. The personal care absorbent article of claim 12 or 16 wherein said article is a diaper, a training pant, a sanitary napkin, an incontinence device or a bandage.

18. A surgical gown wherein at least a portion of said surgical gown comprises the multilayer film of one of claims 1 to 11.

19. A surgical gown wherein at least a portion of said surgical gown comprises the laminate of claims 13 to 15.

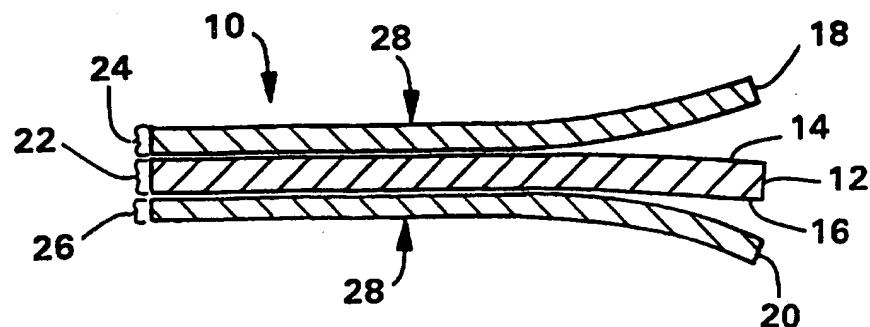
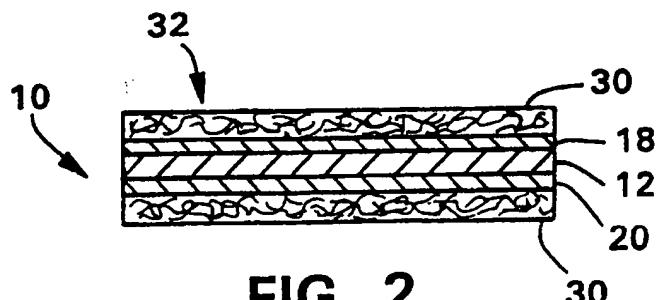
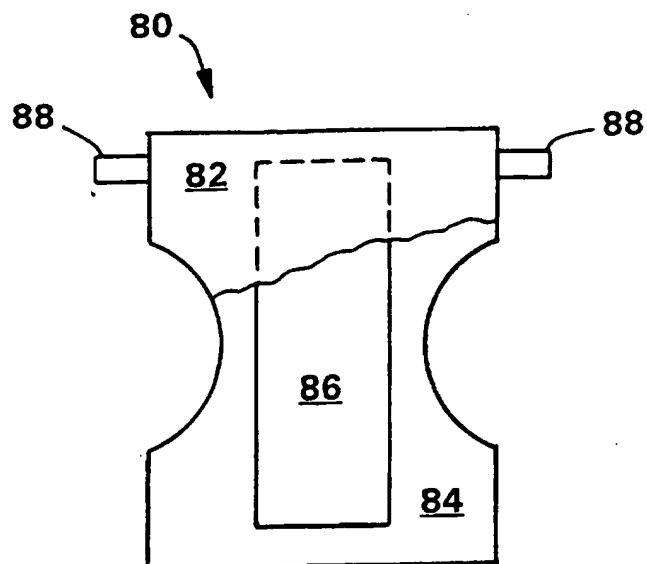
20. A surgical drape wherein at least a portion of said surgical drape comprises the multilayer film of one of claims 1 to 11.

21. A surgical drape wherein at least a portion of said surgical drape comprises the laminate of claims 13 to 15.

22. A article of clothing wherein at least a portion of said article of clothing comprises the multilayer film of one of claims 1 to 11.

23. An article of clothing wherein at least a portion of said article of clothing comprises the laminate of claims 13 to 15.

1 / 2

**FIG. 1****FIG. 2****FIG. 4**

2 / 2

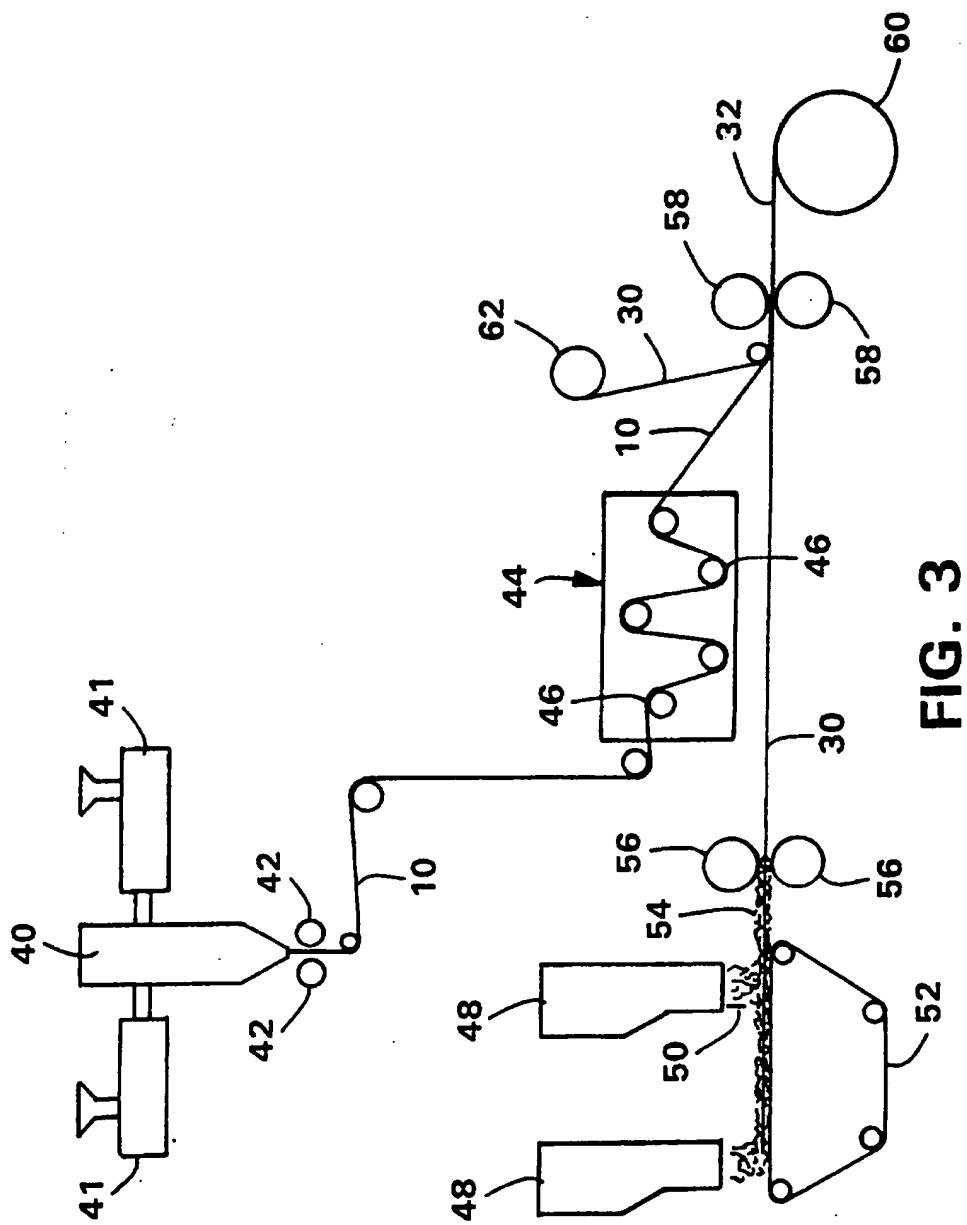


FIG. 3

PCT

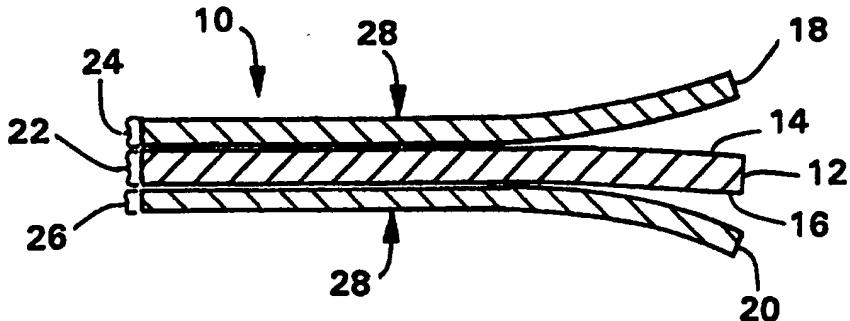
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<b>(71) Applicant:</b> KIMBERLY-CLARK CORPORATION [US/US]; 401 North Lake Street, Neenah, WI 54956 (US). <b>(72) Inventors:</b> MCCORMACK, Ann, Louise; 1265 Poplar Grove Lane, Cumming, GA 30131 (US). HETZLER, Kevin, George; 440 Powers Court Avenue, Alpharetta, GA 30201 (US). <b>(74) Agents:</b> LEACH, Nicholas, N. et al.; Kimberly-Clark Corporation, 401 North Lake Street, Neenah, WI 54956 (US).		<b>(88) Date of publication of the international search report:</b> 17 October 1996 (17.10.96)	

(54) Title: LOW GAUGE FILMS AND FILM/NONWOVEN LAMINATES



(57) Abstract

Disclosed herein is a low gauge, multilayer film (10) which may be laminated to other materials such as, for example, fibrous nonwoven webs. The multilayer film may include one or more skin layers (18, 20) which in certain configurations comprise no more than about 15 percent of the overall thickness and in other configurations no more than about 10 percent of the overall thickness of the multilayer film (10). Such films and laminates have a wide variety of uses including, but not limited to, personal care absorbent products, articles of clothing and health care related items such as surgical drapes and gowns.

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## INTERNATIONAL SEARCH REPORT

Int'l Application No  
PCT/US 95/16624

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 B32B7/00 B32B27/12 A61F13/15 A41D13/12 A61B19/08

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 1 453 649 A (CROWN ZELLERBACH CORP) 27 October 1976 see page 1, line 39 - line 75 see page 2, line 64 - line 98 see page 3, line 21 - line 27; example 1 ---	1-7
Y	US 4 734 324 A (HILL DAVID J) 29 March 1988 cited in the application see column 1, line 18 - line 23 see column 1, line 36 - line 53 see column 2, line 7 - line 9 see column 2, line 25 - line 33 see column 3, line 19 - line 20 see column 3, line 62 - line 63 ---	8-11
Y	-/-	8-11

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

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- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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- \*A\* document member of the same patent family

1

Date of the actual completion of the international search

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Date of mailing of the international search report

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## Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,  
Fax (+ 31-70) 340-3016

## Authorized officer

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## INTERNATIONAL SEARCH REPORT

Intell. Int'l Application No  
PCT/US 95/16624

C(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 247 897 A (EXXON CHEMICAL PATENTS INC) 2 December 1987 see example ---	1-7
X	EP 0 329 377 A (COURTAULDS FILMS & PACKAGING) 23 August 1989 see claims 1,4,6,8,9 ---	1-7,9
X	EP 0 347 745 A (PROCTER & GAMBLE) 27 December 1989 see column 2, line 51 - column 3, line 37 see column 4, line 46 - line 50 see column 8, line 28 - line 40 see column 9, line 22 - line 47; figures 1,3 -----	1-3

**INTERNATIONAL SEARCH REPORT**

International application No.

**PCT/US95/16624****Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)**

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1.  Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely:
  
2.  Claims Nos.: because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
  
3.  Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

**Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)**

This International Searching Authority found multiple inventions in this international application, as follows:

A - Claims 1-11, 18, 20, 22  
B - Claims 12, 17 (in its dependency from claim 12)  
C - Claims 13-16, 17 (in its dependency from claim 16), 19, 21, 23

1.  As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
  
2.  As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
  
3.  As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
  
4.  No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

1-11, 18, 20-22.

**Remark on Protest**

The additional search fees were accompanied by the applicant's protest.  
 No protest accompanied the payment of additional search fees.

## INTERNATIONAL SEARCH REPORT

Information on patent family members

Interr. Application No  
PCT/US 95/16624

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